# PATCHOULI OIL EXTRACTION BY USING HYDRO DISTILLATION (LAB SCALE)

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"I declare that this thesis is the result of my own research except as cited references. The thesis has not been accepted for any degree and is currently submitted in candidate of any degree."

Signature :……..

Name of Candidate :……..

Date :……..

## DEDICATION

*Special dedication to my beloved father, mother,*

*brother and sisters……*

## ACKNOWLEDGEMENT

In preparing this thesis, I was in contact with many people, researchers, academicians and practitioners. They have contributed towards my understanding and thoughts. In particular, I wish to express my sincere appreciation to my main supervisor, Miss. Suriyati Binti Saleh for encouragement, guidance, critics and friendship. I am also indebted to FKKSA lecturers for their guidance to complete this thesis. Without their continuous support and interest, this thesis would not have been the same as presented here.

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## ABSTRACT

Patchouli or its scientific name *Pogostemon cablin*, is used in variety of field. For example it is used in Pharmaceutical industry as effective for fungal or bacterial infection and is of great help for insect bite. Patchouli essential oil is mostly used in perfumery industry as product of cosmetic and fragrance oil. The objectives of this research are to study the effect of extraction time and particle size of the leave on the yield of patchouli essential oil. Effects of extraction time and particle size (grinded and non grinded leave) were studied on the yield of patchouli essential oil. The extraction process is performed by using hydro distillation method. Extraction time is varied at 1, 2, 3 and 4 hours and the extraction process is repeated several times on grinded and non grinded patchouli leave. The essential oil obtained, was analyzed by using GC-MS. From the result, the yield of patchouli essential oil is increased as extraction time increased for both grinded and non grinded leave. The highest yield of grinded and non grinded patchouli leave is at four hours in which 1.32% and 0.89% of yield was obtained respectively. From the result also, grinded patchouli leave produces more oil than non grinded patchouli leave.

## ABSTRAK

Patchouli atau nama saintifiknya *Pogostemon cablin*, digunakan di dalam pelbagai bidang. Sebagai contoh ia digunakan dalam industri perubatan dengan berkesan ke atas jangkitan kulat dan bakteria dan membantu mengatasi gigitan serangga. Minyak patchouli digunakan kebanyakannya dalam industri minyak wangi sebagai produk kosmetik dan wangian. Objektif kajian ini adalah untuk mengkaji kesan masa dan ukuran daun ke atas penghasilan minyak patchouli. Kesan masa dan ukuran daun (daun berkisar dan tidak berkisar) dikaji ke atas penghasilan minyak patchouli. Minyak patchouli dihasilkan meggunakan teknik penyulingan air. Masa yang berbeza digunakan iaitu 1, 2, 3, dan 4 jam dan diulang beberapa kali untuk daun berkisar dan tidak berkisar. Minyak yang terhasil akan diuji menggunakan GC-MS. Daripada hasil eksperimen penghasilan minyak patchouli meningkat apabila masa meningkat. Penghasilan tertinggi minyak patchouli bagi daun berkisar dan tak berkisar ialah selama empat jam di mana sebanyak 1.32% dan 0.89% minyak dihasilkan masing-masing. Daripada hasil eksperimen juga daun berkisar menghasilkan lebih banyak minyak daripada daun ttidak berkisar.

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## LIST OF SYMBOLS

| | | |
|---|---|---|
| g | - | Gram |
| °C | - | Degree Celcius |
| atm | - | Atmosphere |
| m | - | Meter |
| X | - | Multiply |
| mm | - | Milimeter |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Introduction

Essential oil or also known as ethereal oil is a concentrated, hydrophobic liquid that contains hundred of aromatic compounds, organic constituents including hormones, vitamins and other natural elements. These compounds are normally found on leaves, stems, flowers, bark, roots or other elements of a plant. Essential oil contains highly volatile components.

From the vast number of species of plant that are known, about 3000 essential oils have been well identified, though only some 150 have been exploited for commercial production. Inside the plant, the oils are stored as micro droplets in glands. The oils needs to diffuse through the wall of the glands, subsequently the droplets spread over the surface of the plant before evaporating and filling the air with perfume.

Essential oil is used for many different reasons and in different ways. They have a profound effect on the central nervous system, relieving depression and anxiety, reducing stress and relaxing. Many essential oils are used in perfumery. It takes many pounds of flowers to construct ounce of essential oil. Moreover essential oil is utilized as aromatherapy which is a form of medicine. Many essential oil often diluted and sometimes the oil is adulterated with synthetic chemicals.

Briefly, essential oil is a liquid that is generally distilled from the leaves, stems flowers, bark roots or other elements of a plant. Most of essential oils are clear and contain the true essence of the plant it was derived from. It is not the same as perfume or fragrance oils as perfume oils are artificially created fragrances or contain artificial substances and do not offer the therapeutic benefits that essential oil offer.

For this particular experiments, patchouli leaves was chosen for extraction of its essential oil. Patchouli essential oil (patchouli oil) obtained from the leaves of a plant of the same name. The scent of patchouli is heavy and strong. Patchouli is characterized as a perennial bushy herb or shrubs with strong large stems and aromatic furry leaves. Its aromatic oils can be found at various parts of the plant including leaves, stalks, branches and roots. Patchouli leaves gives off the characteristic patchouli odor when rubbed. Figure 1.1 below shows patchouli leave.

**Figure 1.1** Patchouli (Pogostemon cablin)

The patchouli oil is processed through various methods of extraction. Basically,

there are several methods to extract the oil. For example steam distillation, supercritical solvent extraction, ultrasonic extraction, conventional method etc. For this study, hydro distillation extraction method will be used to extract the oil from the dried leaves. Hydro distillation is the most common method of essential oil production. This method is the simplest and cheapest method for ease of use (distillation equipment) and install in the region of patchouli production. Hydro distillation process is chosen in order to maximize the profit and lower the cost, while in the same time to produce a high quality of essential oil.

## 1.2 Problem Statement

Nowadays, Malaysian Herbal Industry gaining more attention and exposure from various parties following the changes in government policy. The local herbal industry is growing at annual rate of between 15% and 20% and has a market value estimated at RM2 billion (Malaysian Industrial Development Authority, 2005). Nowadays, in Malaysia, essential oil and oleoresin is gaining popularity, as a herbal medication as it gave a lot of benefits to overcome some diseases. Hence, patchouli essential oil appeared to be a clear commercial value with a large opportunity to be developed and distributed.

However extraction of patchouli oil is still new in chemical industries. There are no method that proven has been to be the most efficient to extract the oil from patchouli leave. There is a number of factors determine the rate of extraction and the quality of a hydro distilled patchouli essential oil. Aside from the plant material itself, most important are time, temperature, pressure, particle size, and the efficiency of the distillation equipment. Patchouli essential oil also is a very complex product, made up from numerous of distinct molecules which come together to form the oils aroma and therapeutic properties. Some of these molecules are fairly delicate structures which can be altered or destroyed by adverse environmental conditions.

Therefore, this research intent to improve the feasibility of hydro distillation process as a method of patchouli essential oil extraction.

## 1.3 The Objective of the Research

The objectives of this research are to study the effect of extraction time and particle size of the leave on the yield of patchouli essential oil.

## 1.4 The Scope of the Research

The scopes of this research are listed as below:-

1. Patchouli leaves are prepared for the experiment. The leaves are divided into two sample which is non grinded sample and grinded sample (1 mm).
2. A hydro distillation unit is set up and used to extract the essential oils from patchouli leaves. The operating condition temperature is set at $100^{O}C$ and 1atm.
3. In this research, the correlation between the effect of extraction time and surface area is being studied.
4. The essential oil is analyzed by using GC-MS to prove that the oil is patchouli essential oil.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Essential Oil

Essential oil is a highly volatile substance isolated by a physical process from an odoriferous plant of a single botanical species. The oil is named after the plant from which it is derived like rose oil or peppermint oil. Such oils were called essential oil because they were thought to represent the very essence of odor and flavor of the plant it is extracted from unlike vegetable oils expressed from nuts and seeds, essential oil are not actually oily. Some essential oil are viscous, others are fairly solid and most are somewhat watery (http://www.essentialwholesale.com/aromatherapy.html). The oil is also known as volatile oils, or essential oils because of their properties that easily evaporated at ambient temperature.

These oils can be free from the leaves and extracted by using a few method like hydro distillation. Another extraction process like enfleurage (extraction by using fat), maceration, solvent extraction, high pressure CO2 extraction, and cold pressing extraction are also used, however distillation is by far the most common method for extraction of essential oil. Younger plants produce more oils than older ones, but old plants are richer in more resinous and darker oils because of the continuing evaporation of the lighter fractions of the oil.

The first step in isolation of essential oils is crushing or grinding of the plant material to reduce the particle size and to rupture some of the cell walls of oil bearing glands. Steam distillation is by far the most common and important method of production, while extraction with cold fat (enfleurage) or hot fat (maceration) are chiefly of historical important. Three different methods of steam distillation are practiced. In the oldest and simplest method, a vessel containing water and the chopped or crushed plant material is heated by a direct flame, and water vapor and volatile oil are recovered by a water cooled condenser. The original method is being replaced by a process in which the plant material is suspended on a grid above the water level, and steam from a second vessel is introduced under the grid. The volatiles are condensed and the oil is separated. In the third process, the vessel containing the plant material on a grid is heated to prevent condensation of steam, so that dry distillation is attained.

In southern France, essential oil was extracted with cold fat long before the introduction of extraction with volatile solvents. This process is applied to flowers that do not yield a large quantity of oil by steam distillation or whose odor is change by contact with boiling water and steam. In this process flowers are spread over a highly purified mixture of tallow and lard and are left for a period varying from 24 hours to 72 hours. During this time, most of the flower oil is absorbed by the fat. The petals are then remove (defleurage), and the process is repeated until the fat is saturated with oil. The final product is called pomade.

In most cases, it is possible to shorten the long enfleurage process by extracting the essential oil using molten fat for one to two hours at a temperature ranging from 45$^{o}$C to 80$^{o}$C (110$^{o}$F to 175$^{o}$F). The fat is filtered after each immersion and after 10 to 20 extraction cycles, pomade is sold as such, or it may be extracted with alcohol to yield the oil residue.

An extraction procedure called expression is applied only to citrus oils. The outer colored peel is squeezed in process, and the oil is decanted or centrifuged to separate

water and cell debris. The method is used for oil of sweet and bitter orange, lemon, lime, mandarin, tangerine, bergamot, and grapefruit. Much oil is produced as a by-product of the concentrated citrus juice industry.

The function of the essential oil in a plant is not well understood. Odors of flowers probably aid in natural selection by acting as attractants for certain insects. Leaf oils, wood oils, and root oils may serve to protect against plant parasites or depredations by animals. Oleoresinous exudations that appear when the trunk of a tree is injured prevent loss of sap and act as a protective seal against parasites and disease organisms. Few essential oils are involved in plant metabolism, and some investigators maintain that many of these materials are simply waste products of plant biosynthesis.

Commercially, essential oil are used in three primary ways: as odorants they are used in cosmetics, perfumes, soaps, detergents, and miscellaneous industrial products ranging from animal feeds to insecticides to paints; as flavours they are present in bakery goods, candies, confections, meat, pickles, soft drinks, and many other food products; and as pharmaceuticals they appear in dental products and a wide, but diminishing, group of medicines.

### 2.1.1 Chemical Composition of Essential Oil

Terpenes, organic compounds consisting of multiples of isoprene units (containing five carbon atoms), are by far the most dominant constituents of essential oil. Terpenes are built up from units of the simple five-carbon molecule isoprene. Both hydrocarbons and oxygenated compounds such as alcohols, aldehydes, ketones, acids, esters, oxides, lactones, acetals, and phenols are responsible for the characteristic odours and flavours.

Essential oil are generally expensive, with prices ranging from several U.S. dollars per kilogram on the low side to several thousand dollars per kilogram. The high

price of the natural oils coupled with their limited availability has encouraged a search for substitutes. Great progress has been made in the synthesis of individual components such as geraniol, citral, linalyl acetate, and the like. These synthetics have been combined with natural oils to extend supplies, and they have also been blended together in an attempt to duplicate the oils themselves. Such reconstituted oils usually lack certain of the odour notes of the natural products, because of absence of trace ingredients, often unidentified, that may be present in the natural oils. They also tend to have a more "chemical" odour, because of trace impurities in the synthetics that are different from the components of natural oils.

## 2.2 Introduction to Patchouli

Pogostemon cablin (patchouli), like many plants within the Lamiaceae, accumulates large amounts of essential oil. Patchouli oil is unique because it consists of over 24 different sesquiterpenes, rather than a blend of different mono-, sesqui- and di-terpene compounds (Fabienne Deguerry *et al.,* 2006). Patchouli essential oil is very important spiritual oil/root in many countries. Patchouli is a fragrant herb with opposite, egg-shaped leaves, square stems, and famous for its essential oil (patchouli oil). Patchouli oil is obtained by steam distillation or $CO_2$-extraction of the dried leaves. It has its origin in South East Asia and thrives in tropical climes. Distant relatives of this plant include lavender, rosemary and many other herb plants associated with more temperate regions. Usually, it grows to around 3 feet in height and has a dark brownish appearance and its flowers are whitish in color. Patchouli grows well in partially shaded areas, hence it can be grown as an intercrop in coconut gardens. The crop is cut two or three times a year, the leaves being dried and packed in bales and exported for distillation of the oil. Patchouli oil is one of the volatile oils which have a good prospect. It is used mostly as material of cosmetic especially in perfume industry. Patchouli was first described by botanists in the Philippines in 1845. Today growing interest in its fragrance has led to patchouli's widespread cultivation throughout tropical Asia.

Patchouli essential oil used to give a base and lasting character to a fragrance in perfumery industry. The dry leaves of patchouli are put on steam distillation to yield an essential oil called the oil of patchouli. Indonesia is the major producer of patchouli oil in the world with an estimated 550 tons per year, which is more than 80% of the world total production (Robbins and Tao, 1983). Currently, India is producing a meagre quantity of patchouli essential oil and most of its domestic requirement is met by importing about 50 tons of pure oil and 100 tons of formulated oil (M. Singh *et al*., 2002). The market world recently needs 1200-1400 tons Patchouli oil per year. 80-90% demand is supplied from Indonesia. The biggest importer is United State of America which needs 210 tons per year. The other importers are United Kingdom, French, Switzerland, Germany, and Dutch. Figure 2.1 shows a patchouli plantation in Indonesia.

**Figure 2.1** Pogostemon cablin industry scale plantation in Central Kalimantan, Indonesia

Patchouli oil is an essential ingredient and used as a 'base' material in perfumery industry. There is no synthetic substitute for patchouli oil, which increases its value and demand in the perfumery market. Consumption of Patchouli oil in the world is about 2000 tones per annum. In India due to increase in chewing tobacco and pan masala industries, consumption has gone up to about 300 tons per annum while the production is below 50 MT. Hence, the country mostly depends on import mainly from Indonesia and on reconstituted oil.

### 2.2.1 Physical and Chemical Properties of Patchouli

Several species of the Labiatae family with patchouli-like odour grow in the tropics, but only the *Pogostemon patchouli* is utilised for the commercial distillation of the oil. The scent of the essential oil is partly due to patchouli alcohol which first isolated in its crystalline form by Gal in 1869 and formulated as $C_{15}H_{26}O$ by Montgolfier (Adusumilli and Gedu, 2005). Patchouli oil is obtained by various distillation techniques of the leaves of patchouli, and is widely appreciated for its pleasant characteristic and long lasting woody, earthy, camphoraceous odor. It is generally believed that the odour of the patchouli oil is mainly due to norpatchouli alcohol and a closely related norsesquiterpene alcohol (Adusumilli and Gedu, 2005). Patchouli oil is hence an important ingredient in many fine fragrance products like perfumes, as well as in soaps and cosmetic products (K. Bauer et al., 1997). The composition of the patchouli oil is complex like many essential oils, but distinct because it consists largely of sesquiterpenes. The sesquiterpene (ϒ)-patchoulol (Figure 2.1) is the major constituent and is the primary component responsible for the typical patchouli note. The oil contains a large number of other sesquiterpenes hydrocarbons such as α-/β-/ϒ-patchoulenes, α-bulnesene, α–guaiene and seychellene, with structures clearly related to (ϒ)-patchoulol and sesquiterpenes with unrelated structures like *trans*-β-caryophyllene, α-humulene and ϒ-curcumene (Figure 2.1), (Fabienne Deguerry *et al.,* 2006).

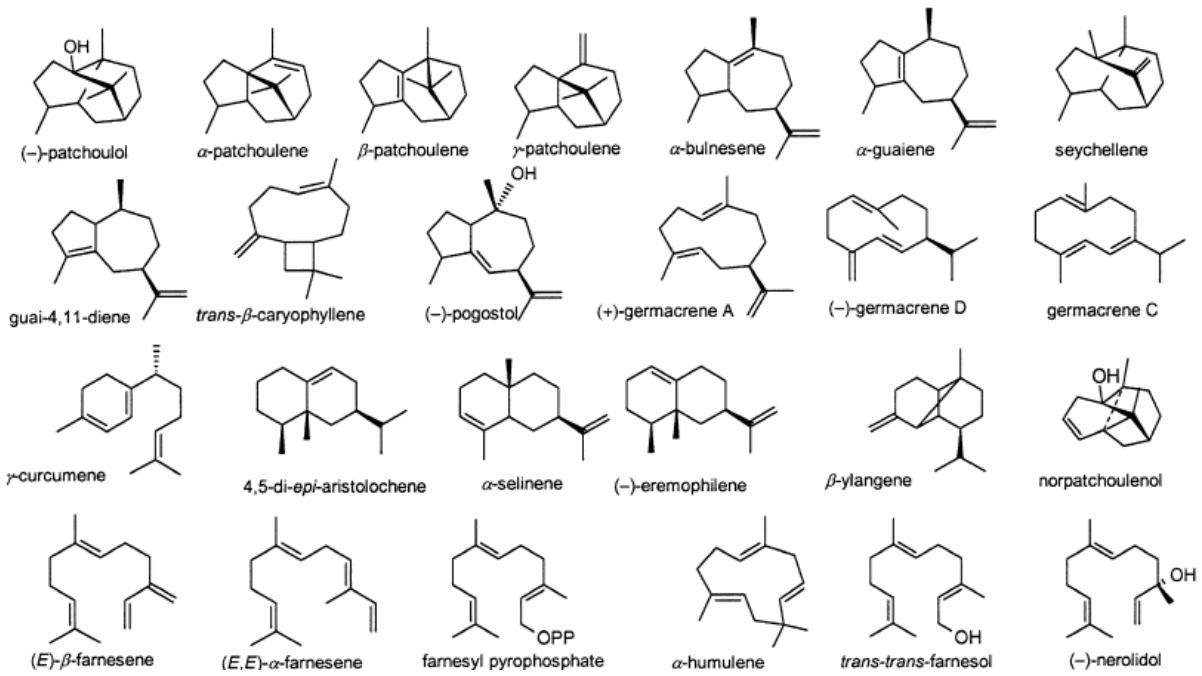


**Figure 2.3** Structure of sesquiterpenes identifed in patchouli oil and/or produced by the recombinant sesquiterpene synthases from patchouli.

At present, patchouli plants are the only commercial source of patchoulol and cost-effective synthetic routes for enantiomeric pure patchoulol have yet to be developed (F. Näf *et al*., 1981). In plants, sesquiterpenes and other volatile secondary metabolites can accumulate on or in the leaves, and often in specialized surface structures such as trichomes. The accumulation and biosynthesis of (¡)-patchoulol and related sesquiterpenes in patchouli leaves has unfortunately received limited attention (W. Henderson *et al*., 1969).

Oil of Patchouli is thick and brownish-yellow tinted green in colour. It contains coerulein,the vivid blue compound found in matricaria, wormwood and other oils. Freshly distilled oil is said to be the best and hence the process is done near the plantations itself. Newly distilled patchouli oil has a fresh, green, slightly harsh aroma. As the oil ages it mellows considerably, becoming sweeter and more balsamic. Other